

# In vitro analysis of femtosecond laser as an alternative to acid etching for achieving suitable bond strength of brackets to human enamel

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**Abstract** This study aims to evaluate the effect of laser irradiation and orthophosphoric acid etching on the shear bond strength (SBS) of orthodontic brackets to enamel. Three groups ( $n=20$ ) of extracted premolar teeth were randomly established depending on the laser treatment performed on the buccal surfaces: (1) no laser (control); (2) Er:YAG laser (2,940 nm, 0.8 W, 100  $\mu$ s/pulse, 10 Hz) and; (3) Ti:Sapphire laser (795 nm, 1 W, 120 fs/pulse, 1 kHz). Each group was divided into two subgroups according to whether 37 %-orthophosphoric acid etching was made after laser irradiation or not. Brackets were randomly luted with Transbond<sup>TM</sup> XT adhesive resin. After 72 h, a SBS test was developed in a universal testing machine (crosshead speed, 0.5 mm/min). Representative specimens from each experimental subgroup were examined by means of scanning electron microscopy. Cement residuals remaining on the premolar surfaces were assessed using the adhesive remnant

index. ANOVA, post-hoc tests for intergroup comparisons, chi-square test and linear regression were run for data analyses ( $\alpha=0.05$ ). After acid etching, SBS values did not differ regardless the laser treatment. When phosphoric acid was not applied, the SBS values of the femtosecond laser group were significantly higher than for the other groups. Femtosecond laser without acid seems to be the most suitable method to improve bond strengths at the bracket/enamel interface, thus avoiding the disadvantages inherent to acid etching.

**Keywords** Femtosecond laser · Enamel · Adhesion · Shear bond strength

## Introduction

The application of 37 %-phosphoric acid for 15 s remains the most common conditioning method for bonding brackets to enamel [1–3]. Despite demonstrating optimal bond strength values [4], the demineralization of the most superficial enamel layer is a potential drawback [5]. As a result, the surface becomes more sensitive to long-term acid attack and caries, mainly in case of incomplete or defective resin impregnation [2, 6].

Exposing enamel to laser irradiation seems to provide some degree of protection against demineralization under acid attack [7]. Laser devices have been used for soft tissue surgery, root end sealing and sterilization; and for altering enamel and dentin surfaces to increase resistance to decay or facilitate bonding of composite resins [8–12]. Nonetheless, whereas some studies report significantly lower bond

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strengths for laser-structured than for acid-etched teeth [2, 4, 13, 14]; others show comparable or even stronger bond strength values for laser treatment [15–17].

The Erbium lasers were specifically introduced in dentistry for cutting enamel and dentin [18, 19]. These lasers emit energy in the wavelength range of 2.6–3  $\mu\text{m}$ . Such interval coincides with the strongest absorption peak of water, which is an important component of dental hard tissues [20]. In particular, the Er:YAG laser (2,940 nm) radiation is strongly absorbed by water and hydroxyapatite.

During the last decade, ultrashort pulsed lasers have been tested as a potential and alternative tool for dental surgery and orthodontics. Sapphire crystals doped with titanium (Ti:Sa) are the main source to produce laser pulses with a duration in the range of the tens and hundreds of femtoseconds. These laser pulses, amplified up to energies of the order of millijoule [21] and conveniently focused on the materials surface, allow the ablation of thin layers with outstanding precision and reproducibility, which may result in much less collateral damage to the adjacent elements than any other thermal, chemical or mechanical process [22, 23]. These lasers have already been used on dental hard tissues [24, 25]. The absorption of ultrashort laser radiation is painless and does not involve vibration or heating. Such qualities make them good candidates for use in dental practice [26].

To our knowledge, there is no previous study comparing the performance of ultrashort lasers with regard to other conventional techniques for improving the bond strength of different orthodontic attachments to enamel surfaces.

Accordingly, the aim of the present in vitro study is to evaluate the influence of two different laser treatments (Er:YAG and ultrashort) and orthophosphoric acid etching on the shear bond strength (SBS) of orthodontic brackets to enamel. The null hypothesis tested is that neither laser treatment nor acid etching, nor the combination of both techniques, influences the SBS of brackets to human enamel.

## Materials and methods

### Sample preparation and storage

A schematic illustration of the preparation of specimens is shown in Fig. 1. Sixty extracted human premolar teeth were collected and stored in a 0.5 chloramine T solution for a maximum of six months after extraction. Exclusion criteria included previously restored premolars and premolars with enamel defects or cracking and delamination of the enamel.

Premolar teeth were examined with an Axio M1 light microscope (Carl Zeiss, Oberkochen, Germany) operating in the dark-field mode. Epiplan  $\times 20$  and  $\times 50$  HD objectives

(Carl Zeiss Vision) were attached to a  $1300 \times 1030$ -pixel digital camera (AxioCam HR, Carl Zeiss Vision). Consistent with the exclusion criteria, the selected premolar teeth were mounted in self-cured acrylic blocks. The buccal surfaces were oriented perpendicularly to the bottom of the molds so that the bonded interfaces were parallel to the force applied during the later SBS test.

Before laser irradiation and acid etching, the buccal crown surface of each premolar was polished for 15 s with fluoride-free pumice slurry, washed for 30 s and dried for 10 s with a moisture-free air spray.

### Experimental groups

Prior to bonding the metal brackets, the premolar teeth were randomly assigned to three groups ( $n=20$ ) depending on the laser treatment to be applied on the enamel surfaces: (1) no laser (control); (2) Er:YAG laser (Fidelis Plus III; Fotona, Ljubljana, Slovenia), and (3) ultrashort pulsed laser (Tsunami; Spectra Physics, Mountain View, CA, USA).

### Laser irradiation

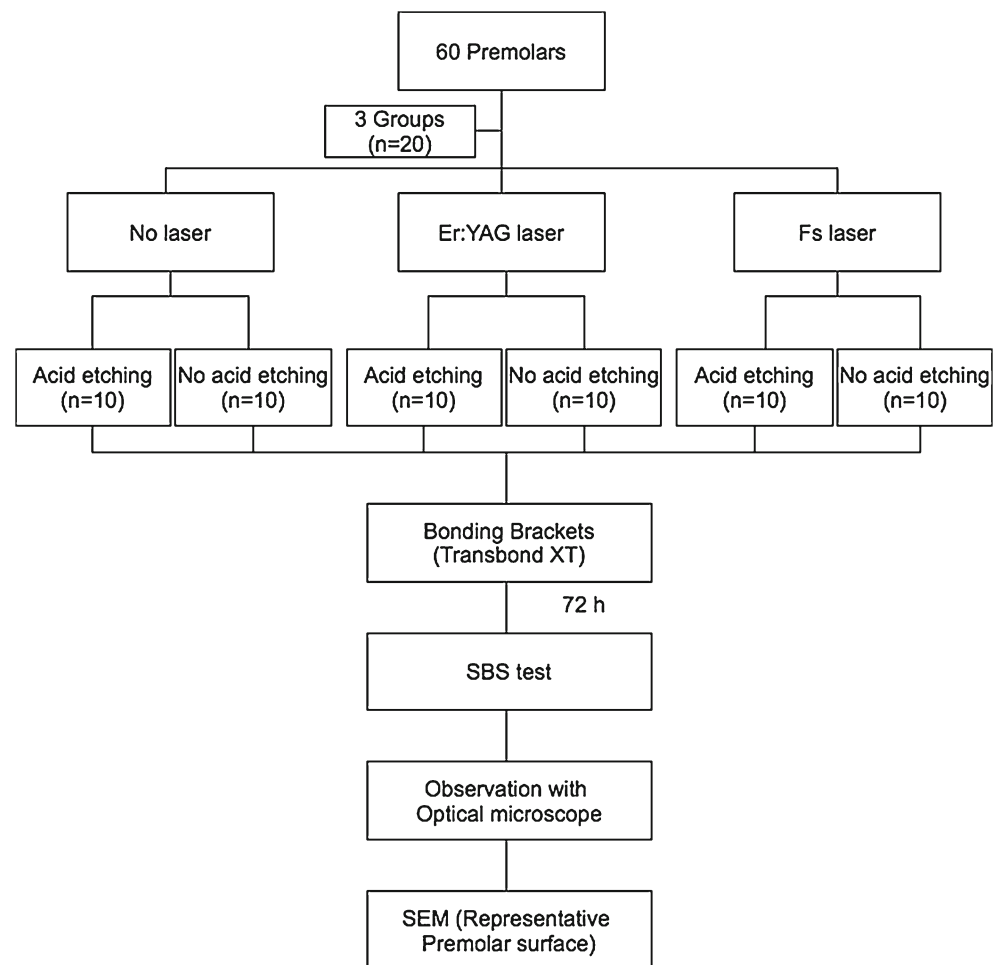
#### *Erbium laser processing*

The Er:YAG laser used in the study emits at a wavelength ( $\lambda$ ) of 2,940 nm. The irradiation was performed under the following conditions: 80 mJ/pulse, VSP (100  $\mu\text{s}$ ), 10 Hz, output power of 0.8 W, focal distance of 10 mm and beam spot diameter of 0.5 mm with a non-contact handpiece (R02). The enamel surfaces were previously moistened to avoid cracking and fusion and were cooled with water spray during irradiation. To simulate as closely as possible actual clinical performance, the laser beam was manually directed without the use of any fixed support.

#### *Ultrashort laser processing*

The laser system consists of a commercial Ti:Sapphire oscillator (Tsunami; Spectra Physics) which provides pulses in the near infrared ( $\lambda=795$  nm) and a regenerative amplifier (Spitfire; Spectra Physics) based on the chirped pulse amplification technique [21] which allows to increase the pulse energy up to 1 mJ. The system delivers pulses with a duration of approximately 120 fs, at a repetition rate of 1 kHz and a maximum output power of 1 W.

The pulse energy is finely controlled by a half-wave plate and a linear polarizer. Neutral density filters were used when further energy reduction was required. The average power of the beam was measured with a thermopile detector (407A; Spectra Physics). The transversal mode is nearly a Gaussian TEM<sub>00</sub> with a 9 mm beam diameter (at  $1/e^2$ ). The laser pulses were focused by means of achromatic doublet

**Fig. 1** Schematic model of the experiment

lens ( $f=100$  mm). With this focusing system the spot size has a diameter of approximately  $12\text{ }\mu\text{m}$ .

The specimens were fixed on a computer-controlled XYZ motorized stage (Micos ES100; Nanotec Electronic GMBH & Co Munich, Germany). The laser pulses impinged vertically on the enamel surfaces. Therefore, the optimum focalization of the pulses on the teeth surfaces was provided by  $Y$  motion and scanning by  $XZ$  motion.

For processing the enamel surfaces a computer code was developed driving the three motors in a way that the three-dimensional surface of each premolar could be homogeneously scanned across the region of interest (ROI). Such ROI-area is in the range of  $15\text{--}40\text{ mm}^2$  depending on the tooth morphology. Since the processing setup does not allow beam motion, the angle between the sample surface and the beam axis must be minimized in order to maximize the absorption of the pulse energy. Otherwise, there would be a substantial difference between the structuring at the apex and at the slopes of the surface. So far, the sample is tilted so that the laser pulses face the flatter surface possible. The scanning pattern was bidirectional.

The enamel was processed in tight focusing conditions. The laser parameters were programmed according to

previous works on ultrashort laser processing of hard dental tissues [23, 25]. The focal length of the lens, the pulse energy ( $0.03\text{ mJ}$ ), the scanning velocity ( $0.5\text{ mm/s}$ ) and the pitch between adjacent scans ( $0.015\text{ mm}$ ) were chosen to generate smoothly overlapping and swallow microstructures.

The teeth samples were laser processed in a saturated vapor atmosphere to preserve the dental tissues from drying. All of the tested specimens were stored in distilled water before and after laser irradiation.

### Acid etching

For each experimental group, half of the specimens ( $n=10$ ) were acid-etched for  $30\text{ s}$  by spreading  $37\%$  phosphoric acid gel (3M™ ESPET™ Scotchbond™, 3M ESPE, St. Paul, MN, USA) on the enamel surface areas where the brackets were to be located (ROI). Afterwards, the buccal enamel surfaces were rinsed with tap water for  $10\text{ s}$  and dried with oil-free and moisture-free air for  $20\text{ s}$  until the enamel had a faintly white appearance as recommended by the manufacturers.

## Bonding procedure

Sixty brackets having micro-etched bases (3M Unitek, Monrovia, CA, USA) were randomly bonded to the premolars' buccal surfaces using a total etch adhesive system to enamel consisting of a combination of a primer and an orthodontic adhesive resin (Transbond™ XT; 3M-Unitek, St. Paul, MN, US). The manufacturer's composition and application mode of the materials used in the experiment are detailed in Table 1.

The adhesive resin was applied to each bracket base (area,  $9.15 \text{ mm}^2$ ) after priming both the tooth and the bracket surfaces [27]. Brackets were then positioned onto the buccal enamel surfaces and pressed firmly with a Hollenback carver to expel the excess adhesive. Each bracket was subjected to a 300-g compressive force using a force gauge (Correx, Berne, Switzerland) for 10 s, after which excess bonding resin was removed using a sharp scaler. Then, the composite was light-cured for 20 s from the occlusal and gingival bracket edges.

The bonding resin was photocured with a LED unit (Bluephase G2; Ivoclar-Vivadent, Schaan, Liechtenstein) emitting in the wavelength range 380–515 nm and a light intensity of  $1,000 \text{ mW/cm}^2$  measured with a built-in radiometer (Bluephase Meter, Ivoclar-Vivadent) which was calibrated every 10 min to ensure consistent light intensity.

## Shear bond strength test

The bracketed teeth were immersed in sealed containers of deionized water and placed in an incubator at  $37^\circ\text{C}$  for 72 h to permit adequate water absorption and equilibration. To conduct the SBS test, the specimens were secured in a jig attached to the base plate of a universal testing machine (Autograph AGS-X 10 KN, Shimadzu, Tokyo, Japan).

The teeth were set at the base of the machine so that the sharp end of the rod incised in the area between the base and the wings of the bracket, exerting a force parallel to the tooth surface in an occluso-apical direction (crosshead speed,  $0.5 \text{ mm/min}$ ). The force required to debond each bracket was registered in newtons (N) and converted into megapascals (MPa) as a ratio of N to the bracket's surface area.

## Failure mode analysis

After the SBS test, each specimen was examined under an optical microscope (Axio M1; Carl Zeiss) at  $\times 50$  magnification to identify the location of the bond failure. The adhesive layers left on the premolar surfaces were assessed by using the adhesive remnant index (ARI), where each specimen was scored according to the amount of material remaining on the enamel surface as follows: 0=no adhesive remaining; 1=less than 50 % of the adhesive remaining; 2=more than 50 % of the adhesive remaining and 3=all adhesive remaining with a distinct impression of the bracket base.

## Scanning electron microscope analysis

Representative premolar surfaces were prepared for scanning electron microscope (SEM) analysis. Samples were dehydrated for 48 h in a desiccator (Sample Dry Keeper Simulate Corp., Tokyo, Japan) and sputter coated with a 10-nm platinum layer in a Polaron E5100 SEM coating unit (Polaron Equipment Ltd., Hertfordshire, England, UK). The morphology of the debonded enamel surfaces was then examined with a variable-pressure SEM (Zeiss EVO MA 25; Carl Zeiss, Jena, Germany).

**Table 1** Manufacturer, main composition and application mode of the materials tested

| Material                         | Manufacturer                | Main components  | Mode/steps of application  |
|----------------------------------|-----------------------------|--|--|
| Scotchbond™ 37 % phosphoric acid | 3M ESPE, St. Paul, MN, US   | 37 % phosphoric acid   | The area where the bracket was to be located was etched with a 37 % phosphoric acid gel for 30 s, rinsed for 15 s, and dried with oil-free and moisture-free air for 20 s until the enamel had a faintly white appearance  |
| Transbond™ XT                    | 3M Unitek; St. Paul, MN, US | Primer: Bis-GMA (Bisphenol A-glycidyl methacrylate), TEGDMA (triethylene glycol dimethacrylate). Adhesive paste: Silane-treated quartz, Bis-GMA, dichlorodimethylsilane reaction product with silica | Primer: air-dry the tooth surfaces thoroughly. Place a thin uniform layer of Transbond™ XT primer on the bracket base and on the tooth enamel surface to be bonded. Adhesive: Apply a thin coat of Transbond™ XT orthodontic adhesive onto the base of each bracket and seat it firmly in place. A minimum amount of composite resin must be utilized to avoid excessive adhesive flash. Scale the excess resin from around the brackets. Photo-cure for 20 s from the occlusal edge and 20 s from the gingival bracket edge |

**Table 2** Mean and standard deviation (SD) of the shear bond strength (SBS) values (MPa) obtained in the experimental groups

| Enamel surface treatments | No laser (control)        | Er:YAG laser               | Ti:Sapphire laser         |
|---------------------------|---------------------------|----------------------------|---------------------------|
| With acid etching         | Mean: 18.6 b B<br>SD: 5.0 | Mean: 20.2 b B<br>SD: 10.9 | Mean: 22.0 b B<br>SD: 7.9 |
| Without acid etching      | Mean: 6.4 a A<br>SD: 2.4  | Mean: 7.8 a A<br>SD: 7.0   | Mean: 22.9 b B<br>SD: 8.3 |

ANOVA test:  $F=9.698$ ;  $P<0.001$ 

Similar lowercase letters in rows and equal capital letters in columns indicate the absence of significant differences

Specific areas of the brackets were also explored to identify possible differences among the experimental groups with respect to the surface topography of such brackets.

### Statistical analysis

Descriptive statistics including means and standard deviations were calculated for the SBS values. Differences in SBS among the experimental groups were examined using ANOVA and Bonferroni multiple comparisons test.

To assess the influence of acid etching and laser surface treatment on SBS, a step-wise multiple linear regression was run, the SBS being the dependent variable. The predictive variable named “Laser” was divided into two dummy variables considering the absence of laser treatment as “control” reference (i.e., Laser 1=Er:YAG against control; Laser 2=ultrashort laser against control). The Determination Coefficient ( $R^2$ ) was taken as the indicator of the model fit.

The ARI scores were analyzed for percentage and frequency of fracture type, and a chi-square test was used to compare acid-etched and non acid-etched samples within each laser treatment group. The ARI scores were categorized as ARI=0–1 vs. ARI=2–3 for statistical comparisons.

All of the statistical analyses were performed using the SPSS v.18 software for Windows (Statistical Package for the Social Sciences, Chicago IL, USA). Significance for all statistical tests was predetermined at  $P<0.05$ . A  $P$  value in

the 0.05–0.10 range was regarded as an indicator of a trend towards statistical significance.

### Results

#### Shear bond strength

Concerning the non acid-etched samples, those treated with ultrashort laser showed statistically higher SBS than those treated with Er:YAG laser or no laser (control), which were statistically similar to each other (Table 2).

When applying acid etching, the three laser treatment groups performed equally concerning the SBS of brackets to human enamel (Table 2).

Acid etching caused no significant effect on SBS in combination with ultrashort laser, whereas within the control and Er:YAG laser groups, acid-etched samples recorded significantly higher SBS values (Table 2).

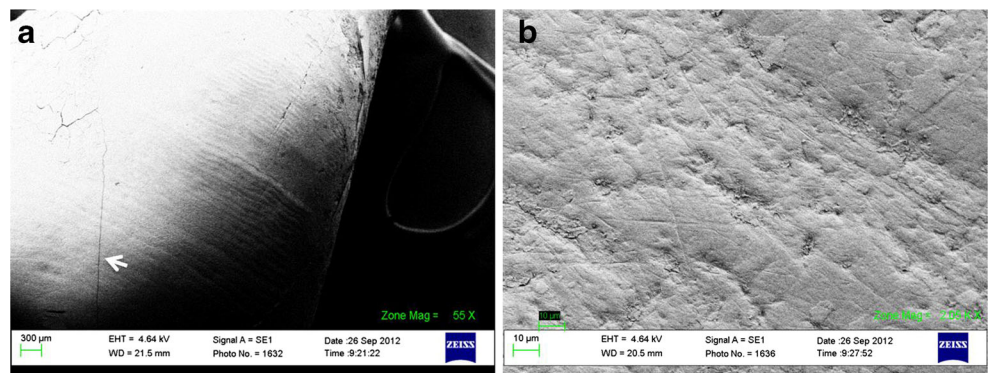
The Multiple Linear Regression model that attempted to predict the model stress values according to acid etching and laser type (MPa) was significant (Chi-square=27.69;  $gl=3$ ;  $P<0.001$ ). Acid etching significantly enhanced the SBS values (7.87 MPa;  $P<0.001$ ). Among the laser systems tested, the ultrashort laser was the only that significantly improved the adhesion as compared to the control group (9.93 MPa;  $P<0.001$ ). The Er:YAG laser tended to increase the SBS values at the adhesive interface, but not significantly (1.46 MPa;  $P=0.55$ ).

**Table 3** Cross-tabulation of the effect of acid etching within the laser treatment groups according to a dichotomous variable generated from the ARI scores (0–1 score vs. 2–3 scores)

| ARI                          | Laser groups      |                 |                             |                 |                             |                 | Total: $n(\%)$                |                 |
|------------------------------|-------------------|-----------------|-----------------------------|-----------------|-----------------------------|-----------------|-------------------------------|-----------------|
|                              | No laser: $n(\%)$ |                 | Er:YAG laser: $n(\%)$       |                 | Ti:Sapphire laser: $n(\%)$  |                 | With etching                  | Without etching |
|                              | With etching      | Without etching | With etching                | Without etching | With etching                | Without etching |                               |                 |
| 0-1 scores                   | 3 (30.0)          | 10 (100.0)      | 8 (80.0)                    | 9 (90.0)        | 2 (20.0)                    | 7 (70.0)        | 13 (43.3)                     | 26 (86.7)       |
| 2-3 scores                   | 7 (70.0)          | 0 (0.0)         | 2 (20.0)                    | 1 (10.0)        | 8 (80.0)                    | 3 (30.0)        | 17 (56.7)                     | 4 (13.3)        |
| Chi-square=10.77<br>$P<0.01$ |                   |                 | Chi-square=0.39<br>$P=0.53$ |                 | Chi-square=5.05<br>$P<0.05$ |                 | Chi-square=12.38<br>$P<0.001$ |                 |



**Fig. 2** SEM micrographs (1 kV) of a debonded sample treated with “no laser/no acid” (**a**  $\times 55$ ; **b**  $\times 2.06$  K). Enamel cracks are labeled in white



### Adhesive remnant index

The ARI scores for the adhesive remaining on the teeth enamel surfaces after debonding are shown in Table 3. Acid etching significantly increased the proportion of specimens in the category ARI=2–3; except for the Er:YAG laser group, in which acid etching had no significant effect.

The laser treatment yielded no significant differences in the ARI scores among non acid-etched samples (chi-square=4.04;  $P=0.13$ ). However, when comparing the distribution of ARI scores depending on the laser type in acid-etched specimens, both ultrashort laser and control groups registered a greater proportion of samples in the category ARI=2–3 than in the category ARI=0–1. The opposite results were observed for the Er:YAG laser group (chi-square=8.42;  $P=0.015$ ).

### SEM observations

Representative SEM images of debonded enamel surfaces after SBS testing are reported in Figs. 2, 3, 4 and 5.

Figure 2 includes micrographs of enamel surfaces treated with “no laser/no acid”. All samples of this subgroup scored “0” in the ARI index (Table 3), showing no adhesive residuals remaining on the teeth surfaces (Fig. 2b). The enamel appears intact, although some microcracks can be observed (Fig. 2a). Such cracks may have occurred due to the metalizing traction or to the SEM vacuum.

**Fig. 3** SEM micrographs (4.64 kV) of a debonded specimen treated with “Er:YAG laser/no acid” (**a**  $\times 55$ ; **b**  $\times 1.97$  K)

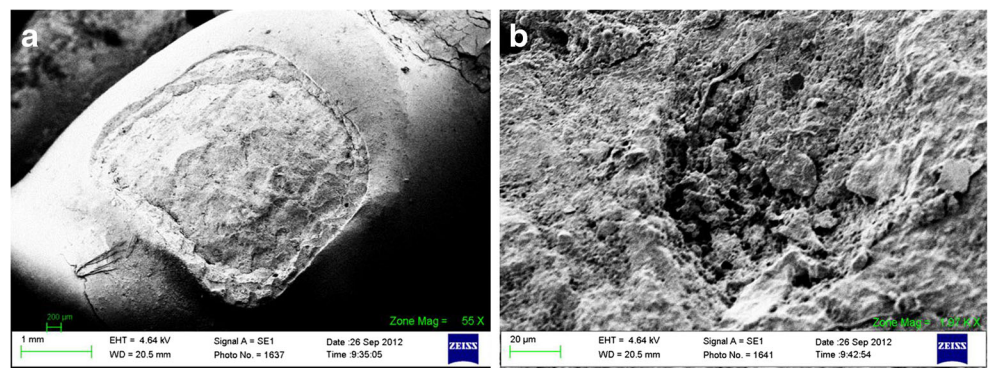


Figure 3 shows micrographs of teeth surfaces etched with “Er:YAG laser/no acid”. In this subset less than 50 % of the adhesive remained on the enamel surface (ARI=1) (Fig. 3a). Sometimes the teeth surfaces contain remarkable peaks and valleys. Although signs of fusion and solidification may be observed, no superficial cracks were identified (Fig. 3b).

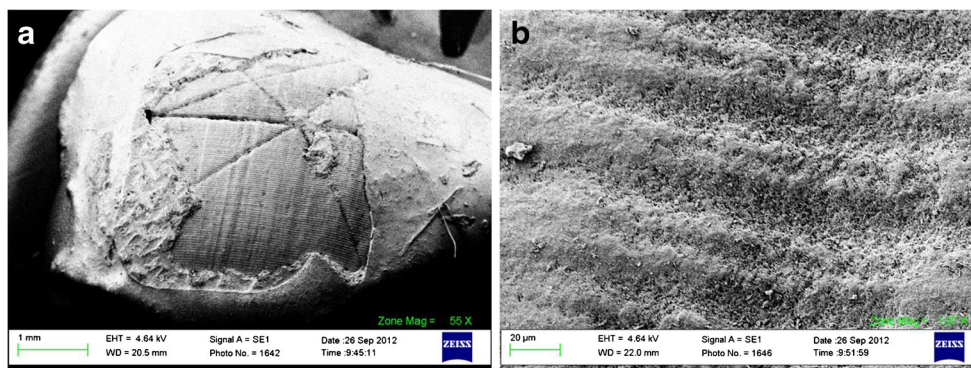
Figure 4 displays micrographs of enamel surfaces treated with “ultrashort laser/no acid” after SBS testing. ARI=1 was the most common failure mode. Less than 50 % of the adhesive remained on the teeth surfaces (Fig. 4a). Figure 4b shows a resin-free zone in which an undulated surface produced by ultrashort laser irradiation can be noticed.

Figure 5 comprises micrographs of enamel surfaces etched “with ultrashort laser/acid”. More than 50 % of the adhesive remained on the teeth surfaces (Fig. 5a). ARI=2 was the predominant failure mode when acid was applied after ultrashort laser. In this case, the phosphoric acid attenuated the pattern left by ultrashort laser processing, although it is still evident (Fig. 5b). Cracks may have occurred due to the metalizing traction or because of the SEM vacuum.

### Discussion

In vitro measurements of the shear debonding forces have been rated as an acceptable methodology to determine future in vivo comparative conditions [14, 27, 28].

**Fig. 4** SEM images (4.64 kV) of a debonded sample treated with “Ti:Sapphire/no acid” (a  $\times 55$ ; b  $\times 1.97$  K)



The results of the current experiment require the rejection of the null hypothesis, as differences among the experimental subgroups were confirmed.

When the teeth enamel surfaces were acid-etched, no significant differences were found for different laser treatments (Table 2). Nevertheless, the bond strength at the bracket/enamel interface significantly decreased in the case of teeth surfaces treated with “no laser/no acid” and “Er:YAG laser/no acid” (Table 2). Findings concerning the SBS values before and after acid application in the “no laser” group were consistent with other studies [15, 28–31]. Acid etching generates microporosities on the enamel surfaces through which the luting resin can penetrate [32]. After polymerizing, the micromechanical interlocking of resin tags within the acid-etched enamel surfaces provides the best achievable adhesion [33]. However, the decalcification of the enamel surface caused by acid etching, facilitates the caries attack [4].

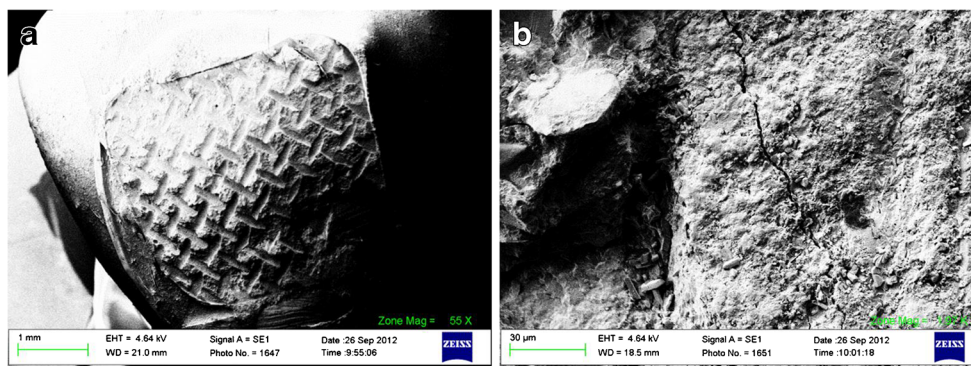
The results of this investigation demonstrate that bonding to Er:YAG laser-treated enamel surfaces provide significantly weaker SBS values than bonding to simply acid-etched surfaces. Nonetheless, the combination of Er:YAG laser plus acid etching produced statistically similar SBS to that of acid-etched surfaces non treated with laser. Such results are in good agreement with former studies [2, 13, 16]. Hibst et al. and Altundasar et al. discovered microcracks on teeth enamel surfaces treated with Er:YAG laser that could be interpreted as a sign of thermal damage [20, 34]. Authors reporting lower bond strengths in related research argued

that microcracks constituted weak regions on the teeth surfaces that give rise to fractures and contaminant filtrations to the tissues [35, 36]. In contrast, some studies find similar or even greater bracket-to-enamel bond strengths when the Er:YAG laser is applied than when the enamel surfaces are just acid-etched [17, 37–39]. Such contradictory results may be attributed to differences among the study protocols. Previous investigations have demonstrated the influence of water flow rate [40], air pressure [41], pulse duration [42], and laser irradiation distances [14] on the ablation rate, efficiency, surface morphology and SBS values. Hence, with the parameters programmed in our experiment it has been evidenced that Er:YAG laser irradiation of human enamel surfaces is not a good alternative to phosphoric acid etching for bonding brackets.

Therefore, either if samples were not laser processed or Er:YAG laser was used, greater SBS values were recorded when the enamel surfaces were acid-etched afterwards. Conversely, the ultrashort laser performed equally in terms of SBS regardless of the use of acid etching (Table 2). The ultrashort laser ability to provide high SBS values without applying phosphoric acid may be due to the micro and nanoroughness produced by laser ablation, which determines an undulated enamel surface texture (Fig. 4b).

To our knowledge, this is the first study on the adhesion of brackets to enamel that uses an ultrashort pulsed laser as a conditioner. In the current experiment, this type of laser has proved to be an appropriate substitute for orthophosphoric acid or Er:YAG laser. With the ultrashort laser, the practical

**Fig. 5** SEM images (4.64 kV) of a debonded specimen treated “Ti:Sapphire/acid” (a  $\times 55$ ; b  $\times 1.97$  K)





absence of thermal load on the remaining dental tissues prevents the formation of microcracks [23] that could impair the adhesion of brackets to enamel. According to our results, the ultrashort laser might replace the phosphoric acid, equaling the SBS and avoiding the adverse effects of acid etching. With this laser, the induction of microstructural changes on the irradiated enamel is minimal; it does not require irrigation and the acoustic disturbance is minimized [23]. Ultrashort pulses could induce thermal fatigue and mechanical damage of dehydrated enamel and dentin [43]. However, as the ultrashort laser generates a plasma-induced ablation, the thermal damage is always lower than that produced with erbium-based laser systems, which emit longer pulses. In our study, the premolar teeth were always hydrated; thereby the adverse effects that may cause the ultrashort laser on dehydrated enamel surfaces were avoided. Although Fig. 5b shows a crack on the enamel surface, which could seem contradictory with the above discussion, this fissure may be due to electron collisions, to the traction generated during metallization or even to the SEM vacuum.

Microscopic observations of the failure sites provided some useful information (Figs. 2, 3, 4, 5). When no acid was applied after the tested laser treatments, more ARI=0–1 values were recorded (Table 3 and Figs. 2, 3, 4) probably because the adhesive does not have enough retention into the enamel causing the bracket debonding. In the surfaces treated with “no laser/acid”; and on enamel surfaces treated with “ultrashort laser/acid” (Fig. 5), most samples recorded ARI scores=2–3 (Table 3). These results are consistent with the literature [14, 15, 28]. Dunn et al. [32] attributed such effect to a blending of the typical pattern of the acid-etched enamel that might prevent the penetration of resin into the enamel surfaces. This can be advantageous for removing the residues after debonding, because less adhesive is expected to be left on the enamel surfaces. However, there is an increased risk of enamel fracture at the time of debonding [28]. When using ultrashort laser, although no differences in SBS were observed between acid-etched and non acid-etched samples, the subsequent application of phosphoric acid might possibly increase the depth of the microretentions. This would make the resin tags deeper and more retentive, leaving more adhesive resin layering on the enamel surfaces.

Clinical trials are necessary to support these conclusions, as ideal laboratory conditions are not common in daily practice [44]. Furthermore, the type of adhesive resin and the strict following of the manufacturers' instructions are also key factors for clinical success [45]. Despite the results obtained with ultrashort lasers in these and other in vitro tests, some drawbacks still preclude their implementation for the clinical practice. Among them, the time to etch the enamel surfaces still remains very long as a result of the

small etching rate per pulse and the low repetition rates available. However some ultrashort laser systems for microstructuring purposes are being developed and even already commercialized reaching repetition rates in the megahertz range what should overcome the lack of competitiveness of this technique. The same can be said with regard to the equipment costs and the dimensions of the laser processing system. In the last years, remarkable steps towards the miniaturization and therefore, the reduction of the investment costs, have been done which foresees a promising future for some applications in the field of odontology.

## Conclusions

Within the limitations of this study, ultrashort pulsed laser seems to be an optimal alternative for bonding orthodontic brackets to enamel. As the adhesion provided by ultrashort laser irradiation and acid etching is comparable, the adverse effects of phosphoric acid can be avoided by using this laser system.

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